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ADVANCED TECHNOLOGY FOR PYROTECHNIC MIXTURES AND MUNITIONS

by

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Studies were made of the raw materials and processing techniques associated with the M18 colored smoke grenades (red, violet, green, and yellow). The findings were that (a) fine particle size dyes can be processed into large particles to reduce dusting during grenade manufacture; (b) coarse particle size dyes and chemicals can be used to manufacture M18 colored smoke grenades, and (c) the percentage of dye used in the M18 grenade can be reduced without affecting the color.

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PREFACE

The work described in this report was authorized under Manufacturing Methods and Technology projects 5731249, 5741249, and 5751249, Advanced Technology for Pyrotechnic Mixtures and Munitions. This work was started in September 1972 and completed in August 1976. The experimental data are recorded in notebooks 8830, 8861, 8919, 9037, and 9189.

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ADVANCED TECHNOLOGY FOR PYROTECHNIC MIXTURES AND MUNITIONS

I. INTRODUCTION.

The purpose of Manufacturing Methods and Technology (MMT) projects 5731249, 5741249, and 5751249 was to conduct a broad study of the pyrotechnic mix used and of all aspects related to loading the M8 and M18 grenades. The study was to include consideration of the basic material specifications, material processing techniques, and equipment related to sizing, screening, handling, and blending of raw materials. The study was also required to better define persistent problems that were evidenced, during manufacture of pyrotechnic white and colored smoke munitions, by the large number of waivers and engineering changes issued both in house at Pine Bluff Arsenal (PBA) and by industrial contractors. The variety and frequency of occurrence of the observed deviations seemed to indicate that the manufacturing processes varied and that the raw materials used failed to meet all the current specifications, although this was not the case. There had to be unknown and therefore uncontrolled critical process variables that contributed to these random product quality variations in addition to known chemical and dye variations leading to product quality variations that were permitted in the specifications to reduce costs.

The goals of the study program were (a) reduction of raw material cost, (b) elimination or better definition of process variables, (c) investigation of improved methods of manufacture, and (d) reduction of safety hazards to operating personnel. It was planned to use the information generated from this project to design new and improved facilities for processing pyrotechnic mixes at PBA.

Project efforts were divided into two major segments–white smoke mix and colored smoke mix. Pine Bluff Arsenal was assigned work on the white smoke mix segment of the program, and EA was assigned work on the colored smoke mix segment. This report does not include the work by PBA, which was treated in a separate PBA technical report, number 17; it covers only the EA studies on colored smoke grenades.

II. CASTABLE COLORED SMOKE MIX.

During the initial stages of this project, a process study of the castable mix being developed by the EA Development and Engineering Directorate was planned. The basic purpose of the castable mix was enabling the direct pouring of the mix into the grenade to eliminate the need of pressing the mix into the grenade. This system could have been automated and thus could have reduced manufacturing costs. In addition, dusting of the castable mix would have been reduced because the mix would not have had to be pressed into the grenade. The initial action undertaken on this task was modification of the EA equipment that had been developed in support of the continuous fluid process for handling the castable mix. Mechanical and electrical modifications were made to the control panel, the electrical drive units, the Merrick feeders, and the load cells. Meanwhile, the development program efforts were directed toward completing the technical data on the castable mix. The latter efforts, although promising, encountered major problems when experimental grenades were subjected to surveillance tests. Change in the technical approach did not solve the problems, and the development group discontinued efforts on the program. This, of course, mandated that the processing studies of castable mix planned under project 5731249 be discontinued and an alternate system be devised to obtain the program goals.

III. STARTER MIX PROCESS.

The development of the plastic-bonded starter mix formulation (PBS-5) is discussed in EATR 4580.[1] The manufacturing process for the PBS-5 was developed under MMT projects 5731249 and 5741249.

Initially, a prototype plastic-bonded starter mix disk machine was developed and tested. This machine volumetrically metered a fluidically controlled quantity of starter mix (14 ± 1 grams) into a plastic mold for making starter disks for use in the M18 colored smoke grenade. This machine worked satisfactorily except for the slow production rate.

Before efforts were made to explore a less expensive and faster manufacturing process for producing the PBS-5 disks, studies to fill gaps in knowledge of the production of the PBS-5 starter mix were performed. Extensive studies of pot life, effect of temperature on curing time, effect of time on fluidity, methods of cleaning production equipment, quality control, sensitivity to cutting, mix segregation, and optimum geometric configuration and weight of the cured disks were conducted.

On the basis of these studies a new system was formulated for manufacturing the PBS-5 starter disk and for automatically inserting the disk into the M18 grenade. The components of this system were modified commercial items.

The system starts with a blending operation. The mix then is poured into 24-inch long cylindrical molds of lay-flat polyethylene tubing and cured in an oven at 70°C or at ambient

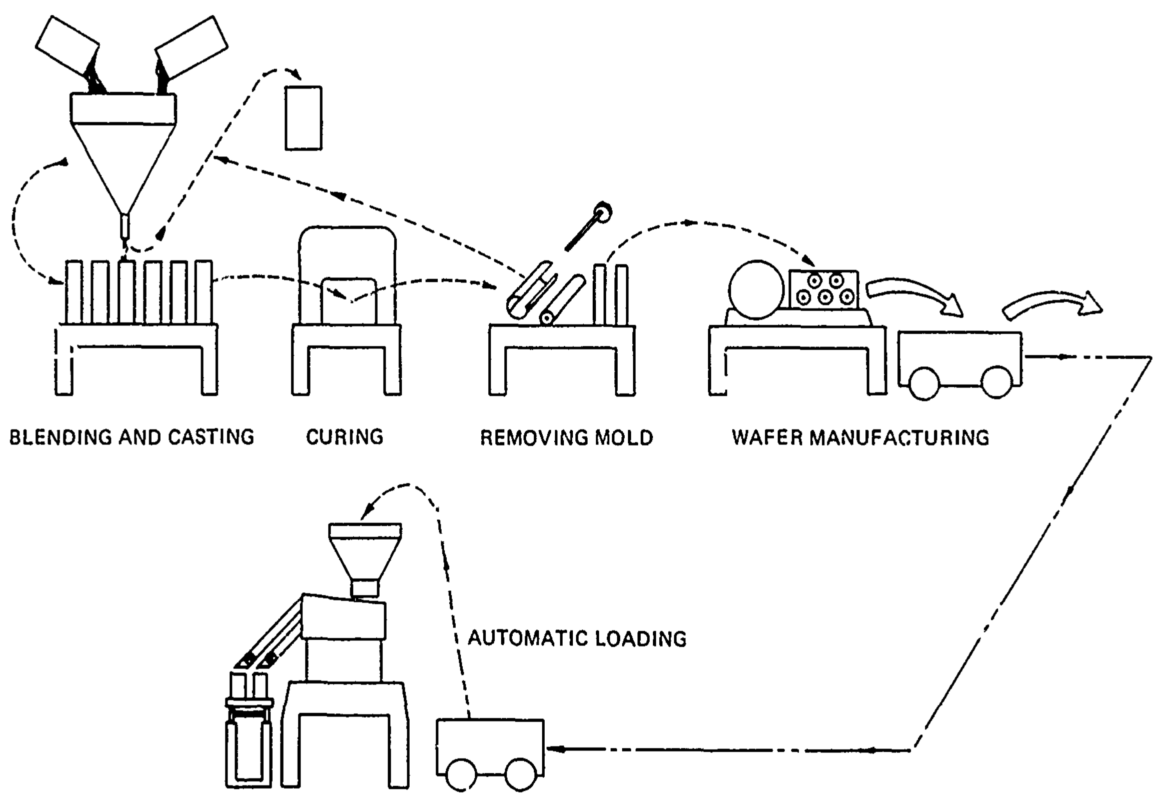

Figure 1. Plastic Bonded Starter Mix (PBS-5) Production Concept, Initial

conditions overnight. The cured cylinder (including plastic tubing) is sliced to form disks (of the desired thickness and weight) that are then put into an automatic dispenser for loading into M18 grenade cans as shown in figure 1. The following pieces of equipment were selected for the proposed manufacturing process:

(1) Blender—an Atlantic Research Corp. Helicone mixer/reactor model 10CV with a mixing capacity of 1 to 10 gal was procured and installed (figure 2).

(2) Slicer—A Berkel model 180D meat slicer, modified to slice three cured cylinders of PBS-5 simultaneously at a rate of 180 disks per minute, was procured.

(3) Automatic dispenser—an automatic PBS-5 loading system was discussed with Syntron Corp. and Cleveland Vibrator Co. A suitable system was designed and proposed, but no procurement was taken because of termination of the program.

The feasibility of manufacturing PBS-5 starter mix was established in this project. However, the substantial increase in material cost ($0.082 per grenade) of the PBS-5 over that of the standard slurry starter mix IV nullified other benefits derived by this process and made the PBS-5 starter mix too uneconomical for use in the M18 colored smoke grenades.

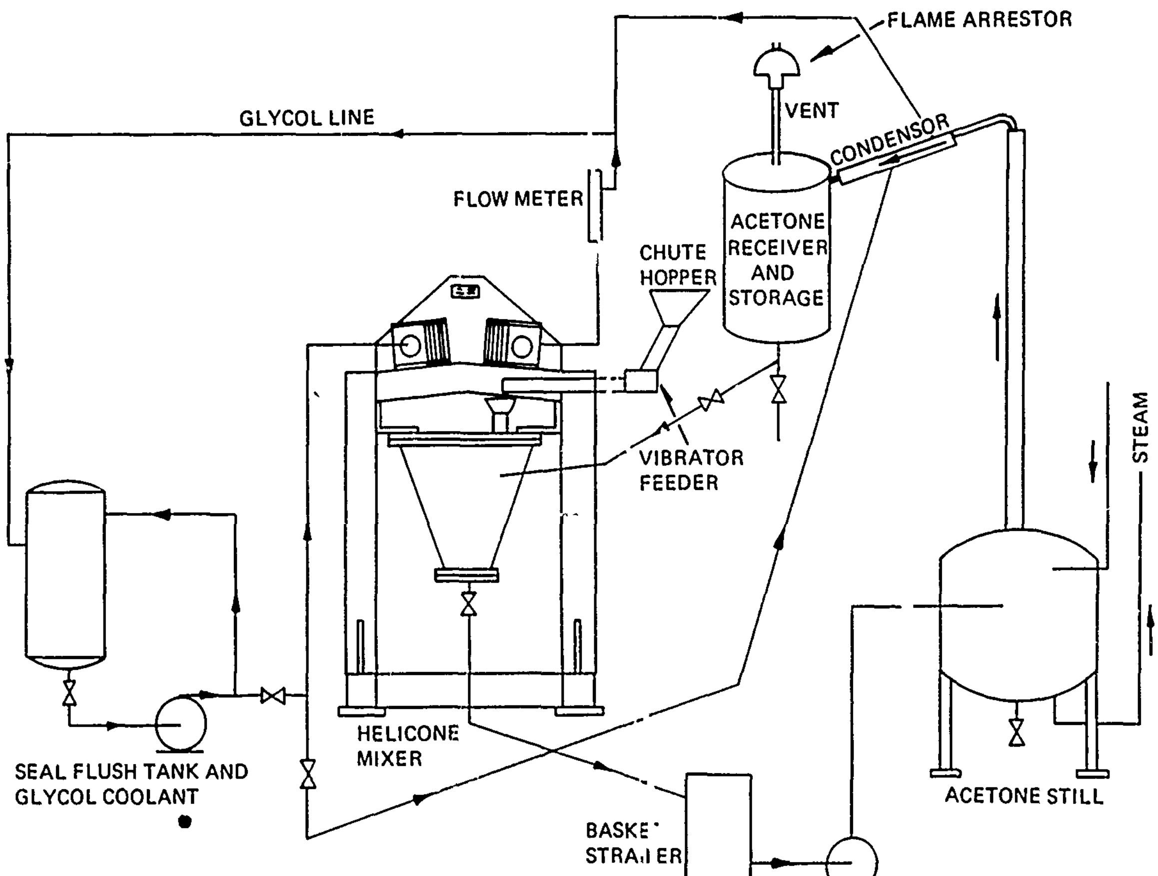

Figure 2. Installation of Helicone Mixer

IV. UTILIZATION OF COARSE MATERIALS.

A. Basic Rationale for Evaluating Coarse Ingredients.

After the termination of the castable colored smoke mix program, the technical approach for achieving the goals of the program was changed. The new approach required the substitution of coarse particle size chemicals and dyes for the extremely fine particle size chemicals and dyes used in the standard mix. It was believed that this change would offer several advantages: It would (a) lower the price of chemicals because the coarse materials were mass produced for other industrial uses, (b) cause less dusting than the extremely fine particles during handling, (c) eliminate the need for the kerosene or acetone that is added to the standard fine ingredients to reduce dusting while handling the mix and pressing grenades. These advantages are explained further in the next section, which also lists the chemical ingredients of a colored smoke mix and describes how these chemicals are currently blended.

B. Standard Materials Production.

1. Standard Materials.

All of the colored smoke mixes used in the M18 grenades are composed of four basic ingredients: a fuel, sulfur; an oxidizer, potassium chlorate; a coolant, sodium bicarbonate; and an agent, dye. Five individual dyes are used in various combinations to produce the four colored smokes: red, violet, green, and yellow. These dyes are:

(1) Dye, disperse red 9: 1-methylaminoanthraquinone[2]

(2) Dye: 1,4-diamino-2,3-dihydroanthraquinone[3]

(3) Dye, solvent green 3: 1,4-di-*p*-toluidinoanthraquinone[4]

(4) Dye, vat yellow 4: 3,4,8,9-dibenzpyrene-5,10-quinone[5]

(5) Dye, benzanthrone: 1,9-benz-10-anthrone[6]

The dyes almost always contain a diluent introduced by the manufacturer to bring the purity of the dyes down to the minimum level required in the dye specifications. Until the year 1972, the type of diluent allowed was unspecified and unknown. Since 1972, the only allowable diluent has been dextrin. However, the exact amount of dextrin used as a diluent is never known and may vary from one dye to another as well as from one manufacturer to another.

2. Standard Processing.

All of the chemicals used in the standard colored smoke mixes (dyes, potassium chlorate, sodium bicarbonate, and sulfur) are very fine powders. If these powders are dry blended, the resultant mix is very difficult to handle in production. The mix does not flow well in equipment for storage, transport, or automatic filling; the mix is extremely dusty, creating an inhalation hazard

requiring protective equipment. In addition, the bulk density is so low that at the M18 grenade production facility at PBA, mix losses of up to 35% were reported at the filling and pressing stations.[7]

For these reasons PBA adopted the current wet blending technique performed in a single planetary mixer. Sufficient acetone is added to the dry ingredients to form a doughlike consistency. As the mixing action continues and the acetone evaporates, the mix is formed into balls or granules 1/8 to 1/2 inch in diameter. The granules are then oven dried to drive off any residual acetone. The resultant mix is free flowing, nondusting, and of a higher bulk density. But, there are problems associated with this mix as well: The mix is more costly to manufacture; a longer blending time is required; the mix must be oven dried; and an expensive solvent, acetone, is used. No attempt is made to recover the acetone expelled into the atmosphere. Should environmental agencies force PBA to monitor and control the level of acetone vapors escaping into the atmosphere, the cost of manufacture would rise even higher.

C. Coarse Chemicals and Dyes.

At the time the program was reoriented from the castable mix approach to the coarse particle size approach, the availability of coarse chemicals and coarse dyes was as described in the following paragraphs.

Sodium bicarbonate samples were received from three suppliers: Church and Dwight Co., Diamond Shamrock Co., and BASF Wyandotte Co. Sodium bicarbonate is manufactured in a wide range of particle sizes. Coarse grades are as readily available as the fine powdered grades. The material chosen for our work was the coarsest offered by Church and Dwight Co., USP grade No. 5. This material is chemically equivalent to O-S-576 sodium bicarbonate.[8] It is very free flowing and any agglomerates present are easily broken during blending.

Sulfur could be ground into any particle size range that one desires. A 16-mesh agricultural grade sulfur distributed by Stauffer Chemical Co. was purchased. This extremely free-flowing sulfur is chemically equivalent to MIL-S-487B but considerably coarser,[9] and it does not agglomerate.

Potassium chlorate was manufactured by two domestic companies: Diamond Alkali Co. and Hooker Chemical Corp. The Hooker Chemical Corp. potassium chlorate was lower in free chlorides and was selected for use. Hooker Chemical Corp. produces only two grades of potassium chlorate, NFX powder and NFX crystal. The NFX crystal is more free flowing than the NFX powder but also forms hard agglomerates when exposed to moist air.

Dextrin was commercially available only as a fine powder. If added to the dye before the dye is granulated, the dextrin acts as an aid in the binder process. We added the dextrin as a fine powder with no significant effect on mix flowability or dusting. The dextrin used was Globe 8032 manufactured by CPC International, Inc. This dextrin met the requirements of MIL-D-3994.[10]

Dyes were available commercially as fine powders. Two dye manufacturers were contacted initially to determine the availability of coarse dyes in the particle size range of US sieve

size numbers 20 through 100. These manufacturers were Atlantic Chemical Co. and American Color and Chemical Co., both of which stated that fine powdery dyes were produced as a result of their process methods. Although the dyes could be additionally processed to make them coarse, neither company currently had the equipment to do so. American Color and Chemical Co. had, in fact, produced coarse granular dyes for sale as recently as 1965.

Commercially available fine powdered dyes were purchased for this project. These dyes were representative of typical process lots and were much higher in purity than specification dyes. The only impurities present in the dyes were byproducts of the formation reactions, and no diluents were added.

D. Studies Conducted With Pellets.

Because coarse dyes were not commercially available, an alternative series of pelletization studies was conducted with red smoke mixes for investigating the feasibility of making tablets or pellets. The purpose of this effort was compaction of the dry, dusty, powdery mix into pellets that would be free flowing without dusting and could be easily loaded into M18 hardware.

Such pellets were produced using Stokes model F and model BB-2 tableting machines. However, the red smoke mix was difficult to feed into the dies of the pelleting machines because the mix bridged in the feed hoppers. This problem was solved in three ways with varying degrees of success:

(1) A vibrating hopper was used.

(2) The mix was kept dry and a free-flowing aid was added.

(3) Smoke mixes were made from fine dyes and coarse chemicals. (This type of mix was the most free flowing.)

Pellets were successfully made using both the dry, powdery, standard smoke mixes and the coarse smoke mixes. The pellets were 3/8 inch in diameter by about 3/8 inch in height. Pellets were made hard enough to retain their integrity under transport through hopper, etc., but were soft enough to have this integrity destroyed under reconsolidation into grenades at 6,500-pounds dead load.

Grenades made with these pellets were examined by means of X-rays and then were function tested. Grenades made with pelletized standard red smoke mix were judged equivalent to those made with powdered standard red smoke mix. The grenades made with pelletized coarse mixes were long burning. This problem was solved later in the program by varying the composition of the mix (reformulation).

It was originally believed that a high-speed pelletizing machine also could be used on a production line. This was based on information from industry that a machine capable of producing 10,000 pellets per minute (1 gram per pellet) was commercially available. At the rate of 10,000 pellets per minute, one machine plus a backup machine would be capable of a production rate of

8,000 grenades per shift. Experimental pelletizing studies determined that the poor flow characteristics of the mix made it unlikely that 10,000 pellets per minute could be produced. Further discussions with machine suppliers indicated that 2,000 pellets per minute was a more realistic estimate. This lower rate meant that five machines would be required on the line to meet the requirement of 8,000 grenades per shift. An analysis of the costs to procure and operate the machines clearly showed that pelletizing of smoke mixes would be an uneconomical approach.

E. Nonavailability of Coarse Particle Size Dyes.

When it became apparent that pelletizing would be extremely expensive, another effort was made to purchase coarse dyes. Letters inquiring into the availability of dyes in the particle size range of US standard sieve size numbers 20 through 100 were sent to approximately 25 producers or suppliers of dyes. The dyes required are listed in section IV. B.1. As a result of these inquiries, a few producers sent samples that they believed might meet the specifications; however, sieve analysis determined all the samples to be too fine in particle size. As a result it was concluded that coarse particle size dyes were not commercially available. The only remaining course of action was processing the dyes from fine particle sizes to the coarse particle sizes required.

F. Dye Granulation Studies.

A method of manufacturing was needed whereby the commercially available fine dyes could be granulated to significantly increase the particle size, reduce dusting, and increase flowability. Although many such granulating processes were available, two processes were selected for the project investigations because of their economic advantages.

1. Chilsonator Studies.

Dyes were granulated at EA using a Chilsonator, manufactured by the Fitzpatrick Co., Elmhurst, Illinois. The Chilsonator is a multistage machine that recycles material to achieve 100% granulation. The first stage of the Chilsonator is the compaction station. The dye moves from a storage hopper by means of a screw feed into the compaction rollers. Two steel rollers rotating in opposite directions compact the dye into thin corrugated sheets. In the second stage of the Chilsonator, these sheets are broken up into granules by a comminuting mill. The granules then fall into the third stage of the Chilsonator where they are sized. A vibrating screen separates the dye into three size increments: product size, oversize, and undersize. The product-size material (i.e., material of the desired size) is discharged into a collection vessel. The oversized granules are recycled through the comminuting mill for regrinding, and the undersized material (fines) is recycled through the compacting rollers.

The Chilsonator used at EA consisted only of the compaction rollers and the controls as shown in the photograph in figure 3. This unit was rented from the Fitzpatrick Co. for 1 week. During this time, 150 to 200 pounds of each of the five dyes were processed into sheets that were later ground into the required granules with a Fitzpatrick model D comminutor. A Sweco 18-inch vibrating screen was used for classifying the granulated dyes and for separating the required particle size ranges.

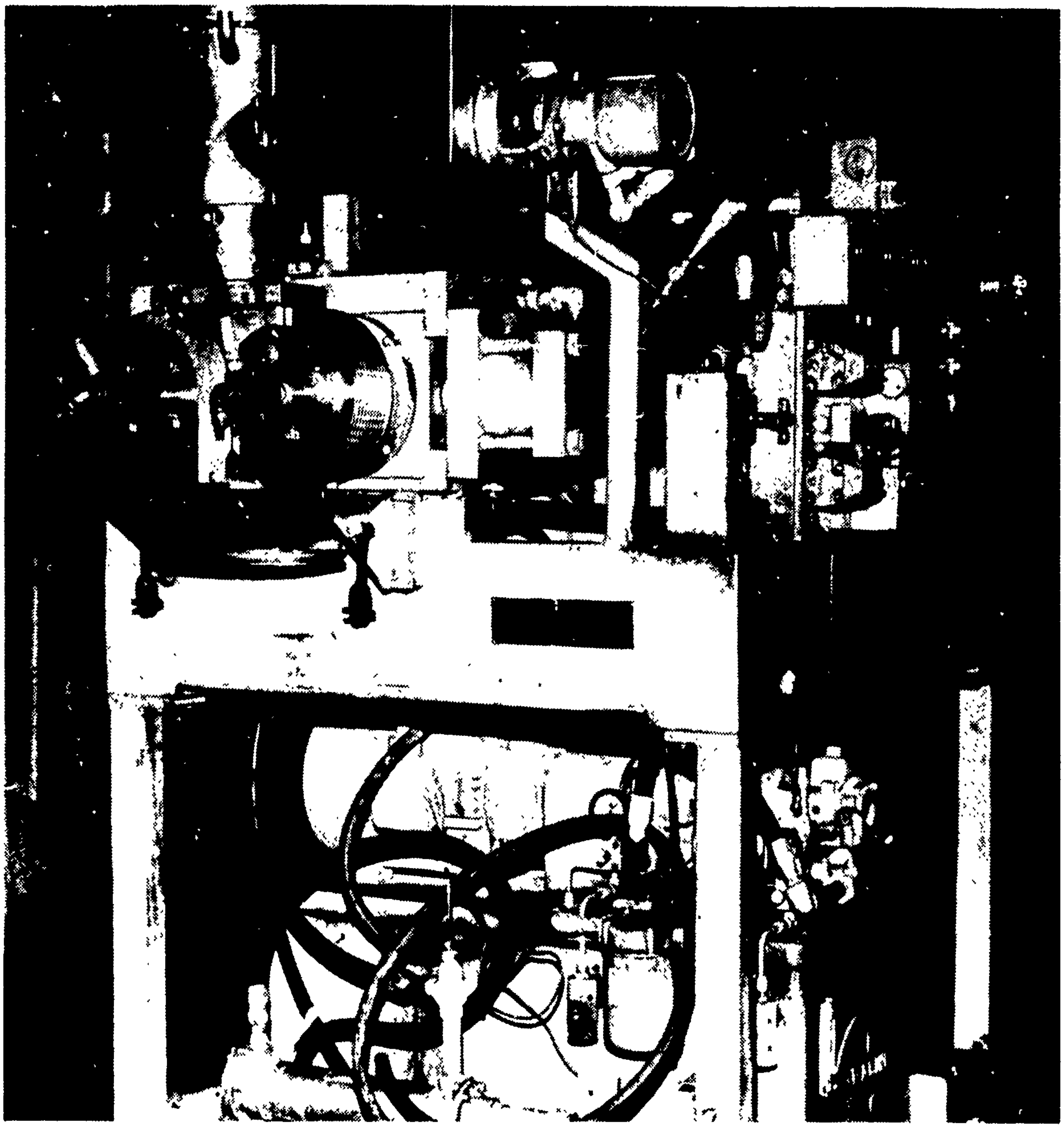

Figure 3. Photograph of Chilsonator

The feasibility of granulating dyes was successfully demonstrated at EA using the Chilsonator. All five of the dyes could be granulated without using any additives, although the 1,4-diamino-2,3-dihydroanthraquinone dye proved difficult to compact. The Chilsonated dyes were reduced to 16- to 100-mesh material (US standard sieve size numbers), and this particle size range was used in the coarse mix studies.

2. Glatt Granulator Studies.

The second process investigated during the course of this project was Glatt dye granulation. The Glatt granulator, manufactured and distributed in the US by Glatt Air Techniques

Inc., Norwood, New Jersey, is commonly used by pharmaceutical industries for granulating powdered medicines. Each of the four dye mixes (red, violet, green, and yellow) was granulated using a model WGS-15 Glatt granulator located at PBA.

In this process a dye with dextrin added according to specification is placed in the processing chamber of the Glatt granulator. Air is forced in at the bottom of the processing chamber, and the dye is fluidized and mixed. While the dye is being fluidized, a fine mist of water is sprayed onto the powder. The water-soluble dextrin (an added diluent allowed by the dye specifications) acts as a binder, and dye granules begin to stick together and to grow. The size of the granules is controlled primarily by the quantity of water added and the cycle time. When the dye granules are of the required size, hot air is introduced into the chamber and the granules are dried (again while being fluidized). Total process time from powder to granules is about 45 minutes for a 20-pound batch. Granules between 20- and 100-mesh (US standard sieve size numbers) are produced with an efficiency for this batch process ranging from 47% to 85% for standardized process controls. Oversize material is pulverized and combined with the undersize material for regranulation in another batch.

Both the Chilsonator and the Glatt granulation processes are capable of agglomerating fine particle size dyes to the coarse particle size required.

G. Experimentation–Coarse Chemicals and Dyes.

Work performed during the pelletizing studies showed that when M18 grenades were made from smoke mixes using coarse chemicals and fine dyes, the grenades were long burning. After granular dyes were made using the Chilsonator and the Glatt granulator, a program was begun to reformulate the four colored smoke blends so that M18 grenades would meet the performance criteria of MIL-G-12326H.[11]

Each colored smoke mix was reformulated by a lengthy series of experiments in which experimental blends were made in the pilot plant, and then the M18 grenade performances were evaluated in field testing. The experimental formulations were blended using a 1/3-ft^3 capacity Patterson-Kelly twin-shell dry blender. The M18 grenades were pressed with a 15-ton capacity Hannefin hydraulic press at a consolidation pressure of 6,200-pound dead load. During blending, loading, and pressing, the coarse mixes were very free flowing and nondusting. No dedusting aid such as kerosene or acetone was added to these mixes.

The results of these reformulation experiments are provided in tables 1 to 4. In all reformulated mixes the chlorate/bicarbonate ratio had to be increased for the grenades to burn within 50 to 90 sec and to emit an acceptably colored smoke cloud. Note that dextrin is included as an ingredient in these formulas. In our experiments dextrin was added as a separate ingredient, but it could also be added with the dyes as a diluent or as a process aid as in the Glatt granulation process.

Experiments conducted during the time period of this project both at EA and PBA showed that the amount of dextrin present as a diluent in a dye affects the burning characteristics of the smoke mix. So, in a sense, the burn time of a grenade is dependent on the dextrin content of the dye from which the grenade is made. Therefore, it is important that the effect of dextrin

Table 1. Project 5751249–Experimental Red Grenades

Formula No.	Dye*		KClO$_3$		NaHCO$_3$	Sulfur	Dextrin	Burn time	Lag time
	Type	Amount	Type	Amount					
		%		%		%		sec	
61	Chilsonator	35	NFXC	31	22.3	8.7	2.9	65	3-4
62	Chilsonator	35	NFXC	31	22.3	8.7	2.9	65	3-4
65	Chilsonator	35	NFXC**	29	24	9	3	71	10
66	Chilsonator	35	NFXC**	29	24	9	3	66	5
68	Chilsonator	35	NFXC**	28	25	9	3	95	15-20
71	Chilsonator	34	NFXC**	29	24	9	4	73	6
72	Chilsonator	32	NFXC**	29	24	9	6	76	6
77	Chilsonator	32	NFXC	29	24	9	6	79	9
90	Glatt granulator	36.88	NFXC**	29	22	9	3.12	85	4
91	Chilsonator	34	NFXC**	29	22	9	6	68	4
96	Glatt granulator	36.88	NFXC	30	21	9	3.12	73	5
106	Chilsonator	32	NFXC	30	21	9	8	70	7-9
107	Chilsonator	34	NFXC	28	23	9	6	93	15
108A	Chilsonator	32	NFXC	30	23	9	6	70	4
117	Chilsonator	36	NFXC	26	21	10	7	106	8
127	Chilsonator	35	NFXC	28	21	10	6	78	5
128	Chilsonator	32	NFXC	30	23	10	5	65	5
129	Chilsonator	32	NFXC	30	23	8	7	78	5
131	Chilsonator	35	NFXC	31	20	9	5	59	5
132	Chilsonator	37	NFXC	27	20	9	7	94	30
133	Chilsonator	36	NFXC	27	22	9	6	107	35

*Dye: disperse red 9.
**NFX crystal KClO$_3$ comminuted through 50-mesh screen.

amount versus burn time be studied and that the exact amount of dextrin used as a diluent or process aid be known and controlled. It is not sufficient to eliminate dextrin from the smoke mixes altogether, as water vapor, the combustion product of dextrin, plays an important role in dissemination of the dye aerosol. For these reasons, dextrin has been included as an ingredient in the formulas, and optimum percentages were selected for each color of smoke.

On the basis of these reformulation tests, an optimum blend was selected for each color of smoke, and larger (20-kg) batches were made using the 2-ft^3 capacity Patterson-Kelly twin-shell dry blender. Grenades made from these optimum formula batches were used to support the required tests discussed in the following section.

H. Testing of Coarse Material Grenades.

To insure that the coarse materials would be an acceptable alternative to the standard materials specified in the technical data package, it was decided to conduct tests for substantiating their acceptability. The testing deemed necessary was established through discussions with various members of the EA Configuration Control Board. The selected tests were functional tests and hazard classification tests. The formulations of the smoke mixes that were used in both types of test are shown in table 5.

Table 2. Project 5751249–Experimental Violet Grenades

Formula No.	Dye[a]			$KClO_3$		$NaHCO_3$	Sulfur	Dextrin	Burn time	Lag time
	Type	V	R	Type	Amount					
		%			%		%		sec	
69	Chilsonator	([b])	([b])	NFXC[c]	27	23	9	6	66	5-10
70	Chilsonator	([b])	([b])	NFXC[c]	26	24	9	6	79	15
82	Chilsonator	28	7	NFXC[c]	28	22	9	6	54	5
83	Chilsonator	28	7	NFXC[c]	29	21	9	6	46	4
86	Chilsonator	28	7	NFXC[c]	27	22	9	7	64	4
87	Chilsonator	28	7	NFXC[c]	26	24	9	6	74	19
92	Glatt granulator	([d])	([d])	NFXC[c]	28	22	9	2.07	45	4
97	Glatt granulator	([d])	([d])	NFXC	28	22	9	2.07	46	4
100	Glatt granulator	([d])	([d])	NFXC	27	23	9	2.07	47	3
101	Chilsonator	28	7	NFXC	27	23	9	6	58	5
108	Glatt granulator	([d])	([d])	NFXC	26	24	9	2.07	59	3-4
115	Chilsonator	30.4	7.6	NFXC	24	21	10	7	113	7
116	Chilsonator	30.4	7.6	NFXC	24	25	8	5	165	([e])
124	Chilsonator	28.8	7.2	NFXC	25	23	10	6	80	8
125	Chilsonator	28	7	NFXC	27	23	10	5	66	5
126	Chilsonator	28	7	NFXC	27	23	8	7	64	5
130	Chilsonator	23.6	7.4	NFXC	26	21	9	7	69	8

[a] Dyes: V = 1,4-diamino-2,3-dihydroanthraquinone; R = disperse red 9.
[b] Total amount of V and R dyes combined = 35%; individual amounts unknown.
[c] NFX crystal $KClO_3$ comminuted through 50-mesh screen.
[d] Total amount of V and R dyes combined = 38.93%; individual amounts unknown.
[e] Datum not recorded.

1. **Functional Tests.**

EA special test No. 75-11 was conducted by the Technical Support Directorate at EA. Details of the test are summarized in table 6. The following tests of experimental M18 grenades in standard packaging were carried out:

(1) Rough handling tests:

(a) High-altitude, low-pressure
(b) Free fall (5-foot)
(c) Transportation vibration
(d) Transportation shock

(2) Simulated environmental tests:

(a) Arctic
(b) Tropic
(c) Desert
(d) Cyclic

Table 3. Project 5751249–Experimental Green Grenades

Formula No.	Dye[a] Type	Dye[a] G	Dye[a] B	Dye[a] Y	$KClO_3$ Type	$KClO_3$ Amount	$NaHCO_3$	Sulfur	Dextrin	Burn time	Lag time
		%				%	%			sec	
75	Chilsonator	26	7	3	NFXC[b]	30	21	10	3	75	6
76	Chilsonator	25	7.5	3.5	NFXC[b]	29	21	9	5	77	6
78	Chilsonator	25	7.5	3.5	NFXC[b]	29	20	9	6	74	7
79	Chilsonator	26	7	3	NFXC[b]	28	21	9	6	84	8
88	Chilsonator	23.8	6.8	3.4	NFXC[b]	29	22	9	6	67	4
89	Chilsonator	23.8	6.8	3.4	NFXC[b]	31	20	9	6	72	5
94	Glatt granulator	– (c)	36.73 (c)	– (c)	NFXC[b]	29	21	9	4.27	73	7
95	Chilsonator	24.5	7.0	3.5	NFXC[b]	29	21	9	6	71	5
99	Chilsonator	24.5	7.0	3.5	NFXC	30	20	9	6	78	4
104	Chilsonator	24.5	7.0	3.5	NFXC	31	19	9	6	71	3-4
105	Chilsonator	24.5	7.0	3.5	NFXC	29	21	9	6	79	3-5
112	Chilsonator	25.9	7.4	3.7	NFXC	27	19	10	7	104	14
113	Chilsonator	24.5	7.0	3.5	NFXC	27	23	9	6	91	9
114	Chilsonator	23.1	6.6	3.3	NFXC	31	23	8	5	79	5
121	Chilsonator	24.5	7.0	3.3	NFXC	32	21	9	5	67	7
122	Chilsonator	24.5	7.0	3.5	NFXC	30	20	10	5	70	6
123	Chilsonator	24.5	7.0	3.5	NFXC	30	20	8	7	70	6
134	Chilsonator	25.2	7.2	3.6	NFXC	28	19	10	7	71	6

[a] Dyes: G = solvent green 3; B = benzanthrone; Y = vat yellow 4.
[b] NFX crystal $KClO_3$ comminuted through 50-mesh screen.
[c] Total amount of G, B, and Y dyes combined = 36.73%; individual amounts unknown.

Table 4. Project 5751249–Experimental Yellow Grenades

Formula No.	Dye[a] Type	Dye[a] Y	Dye[a] B	$KClO_3$ Type	$KClO_3$ Amount	$NaHCO_3$	Sulfur	Dextrin	Burn time	Lag time
		%			%	%			sec	
73	Chilsonator	12.5	22.0	NFXC[b]	22	29	8.5	6	66	12
74	Chilsonator	12.5	22.0	NFXC[b]	23	30	8.5	4	59	10
80	Chilsonator	11.8	20.7	NFXC[b]	22	29	8.5	8	86	8
81	Chilsonator	11.8	19.4	NFXC[b]	23	31	8.5	7	77	6
84	Chilsonator	11.8	20.7	NFXC[b]	24	30	8.5	5	55	4
85	Chilsonator	11.8	20.7	NFXC[b]	25	28	8.5	6	50	3
93	Glatt granulator	(c)	(c)	NFXC[b]	24	30	8.5	3.11	74	5
98	Chilsonator	11.8	20.7	NFXC	24	30	8.5	5	63	7
102	Chilsonator	11.8	20.7	NFXC	23	31	8.5	5	73	6
103	Chilsonator	11.8	20.7	NFXC	25	29	8.5	5	54	4-5
109	Chilsonator	12.9	22.6	NFXC	21	28	9.5	6	85	14
110	Chilsonator	11.8	20.7	NFXC	21	32	8.5	6	107	30
111	Chilsonator	11.4	20.1	NFXC	25	32	7.5	4	63	6
118	Chilsonator	12.5	22.0	NFXC	23	27	9.5	6	61	5
119	Chilsonator	11.8	20.7	NFXC	24	30	9.5	4	61	6
120	Chilsonator	11.8	20.7	NFXC	24	30	7.5	6	64	6
135	Chilsonator	11.8	20.7	NFXC	23	32	8.5	4	68	8

[a] Dyes: Y = vat yellow 4; B = benzanthrone.
[b] NFX crystal $KClO_3$ comminuted through 50-mesh screen.
[c] Total amount of Y and B dyes combined = 34.39%; individual amounts unknown.

Table 5. Formulas for Bulk Mix and Grenades, EA and NASA Tests

Color	Dye*					$KClO_3$	$NaHCO_3$	Sulfur	Dextrin
	B	G	R	V	Y				
	%								
Red	–	–	32	–	–	30	23	9	6
Yellow	20.7	–	–	–	11.8	24	30	8.5	5
Green	7.0	24.5	–	–	3.5	30	20	9	6
Violet	–	–	7	28	–	27	23	9	6

*Dyes: B = High-purity dye, benzanthrone; G = High-purity dye, solvent green 3; R = High-purity dye, disperse red 9; V = High-purity dye, 1,4-diamino-2,3-dihydroanthraquinone; Y = High-purity dye, vat yellow 4.

Table 6. EA Special Test No. 75-11, Project 5751249

Test	Box Nos.*	Total No. of grenades	Standard grenades		Experimental grenades	
			Number	Problems	Number	Problems
Rough handling	1, 2, 4, 11, 13, 14, 17, 20	128	64	18	64	7
Cold soak	7, 9	32	16	5	16	3
Tropic storage	3, 18	32	16	0	16	0
Desert storage	5, 15	32	16	0	16	4
Cyclic storage**	12, 22	32	8	0	24	9
Arctic storage	6, 16	32	16	4	16	2
Hot cyclic**	10, 21	32	8	2	24	2

*Boxes used in the test were consecutively numbered 1 through 22. Boxes 1 through 20 contained 8 experimental and 8 standard grenades. Boxes 21 and 22 each contained 16 experimental grenades scheduled for a visibility test. Because of an error, boxes 21 and 22 were used in the primary tests listed in the table, and boxes 8 and 19 were used in the visibility test.

**Original plans were to subject 16 standard and 16 experimental grenades to each of these tests; however, because of an error, 8 standard and 24 experimental grenades were tested.

(3) Design efficiency assessment trials:

(a) Hot cyclic
(b) Cold soak

(4) Smoke color identification tests

The results of the EA tests show there was little difference between the experimental coarse material grenades and the standard M18 grenades. The only problem arising from the test was that a high number, nine, of experimental grenades exploded. This problem was reviewed, and it was determined that the coarse materials in the mix did not cause the malfunction. Excessive starter mix applied during manufacture reduced the size of the center hole and resulted in excessive pressure causing all the grenades to explode, as observed, simultaneously with the detonation of the fuzes. This conclusion was further substantiated by the fact that during the developmental program 1200 grenades were tested without incident.

2. Hazards Classification Tests.

Tests were conducted by the General Electric Co. at the NASA National Space Technology Laboratories, Bay St. Louis, Mississippi, for the purpose of providing classification testing in accordance with US Army Technical Bulletin TB 700-2, "Explosives Hazard Classification Procedures, change 1." The results of these tests were reported in Edgewood Arsenal Contractor Report EM-CR-76104.[12] The following experimental bulk materials and end-item munitions were tested:

(1) Red smoke mix

(2) Violet smoke mix

(3) Green smoke mix

(4) Yellow smoke mix

(5) M18 red smoke grenades

(6) M18 violet smoke grenades

(7) M18 green smoke grenades

(8) M18 yellow smoke grenades

The classification of bulk pyrotechnic compositions is accomplished by the evaluation of test data obtained on their ease of ignition and on their stability prior to shipping and handling. The specific tests conducted for this study were (a) detonation, (b) ignition and unconfined burning, (c) thermal stability, (d) impact sensitivity, and (e) card gap.

The evaluation of pyrotechnic end-item munitions is accomplished by the evaluation of test data designed to determine their tendency to propagate munition functioning from one shipping case to another and by evaluation of the reaction resulting from burning the munitions in an intense fire. The following specific tests were conducted: (a) Detonation Test A, (b) Detonation Test B, and (c) External Heat (Open-Flame) Test C.

In all US Army TB 700-2 tests except one, the impact sensitivity test for bulk materials, the four experimental bulk compositions and end-item munitions yielded essentially identical test results as the standard M18 smoke formulations. Results of the impact sensitivity tests reveal that all four experimental pyrotechnic mixes exhibited a greatly increased sensitivity toward impact.

I. Physical and Chemical Analysis of Materials.

A total of 14 dye and chemical samples was analyzed by the Product Assurance Directorate (PAD) for selected physical and chemical characteristics. Eleven samples were various types of dyes; the remaining three samples were potassium chlorate, sulfur, and sodium bicarbonate. The results of these analyses, furnished in tables 7 to 11, were used in establishing new material specifications for the coarse chemicals and dyes.

Table 7. Analysis of Dyes (As Received), 23 January 1976

Color	Sample No.	Purity (average)	Volatile matter	Density	Particle size No.						Marcol 52	Supplier	Lot No.
					20	50	60	100	200	325			
		%	%	gm/ml	% passing						%		
Red 9	1	95	0.1	0.16	–	–	100	100	100	–	–	ACC	9627
	1A	97	–	–	–	–	97	90	70	–	–	AtC	49025-18
	Specification[a]	90	2.5	.35	–	–	–	–	–	–	–	–	–
Yellow 4	3	84	2.6	.20	100	100	–	–	–	100	–	AtC	V100-495-6-1
	Specification[b]	80	1.0	–	100	97	–	–	–	40	2.0	–	–
Green 3	5	99	.1	.46	100	100	–	–	–	88	–	AtC	600395-34-2
	Specification[c]	90	2.5	.38	100	97	–	–	–	40	–	–	–
1,4-diamino-2,3-dihydro-anthraquinone	7	97	.2	.55	–	–	87	67	48	–	–	AtC	69100-212
	Specification[d]	70	2.5	.35	–	–	97	90	70	–	–	–	–
Benzanthrone	9	90	.6	–	100	100	–	–	–	100	–	AtC	1R55000-11-2
	Specification[e]	77	1.0	–	100	97	–	–	–	40	2.0	–	–

ACC = American Color and Chemical Co.; AtC = Atlantic Chemical Co.
[a]MIL-D-3284C.
[b]MIL-D-0050029C (MU).
[c]MIL-D-0050029C (MU).
[d]MIL-D-3668B (MU).
[e]MIL-D-0050074C (MU).

Table 8. Analysis of Agglomerated Dyes

Sample No.	Color*	Particle size No.						Density	Supplier	Lot No.
		20	30	40	60	100	200			
		% passing						gm/ml		
2	R	87	71	58	46	38	32	0.4[illegible]	ACC	9627
4	Y	89	57	34	16	6	1	.66	AtC	V100'95-6-1
6	G	90	66	47	24	10	4	.64	AtC	600395-34-2
8	V	92	80	64	44	23	12	.62	AtC	69100-212
10	B	82	55	33	16	5	.5	.61	AtC	1R55000-11-2

ACC = American Color and Chemical Co.; AtC = Atlantic Chemical Corp.
*R = red 9; Y = yellow 4; G = green 3; V = 1,4-diamino-2,3-dihydroanthraquinone; B = benzanthrone.

J. Establishment of New Formulas.

Based on the experimentation and testing of coarse chemical smoke mixes conducted under this project, new formulas have been established as summarized in tables 12 to 15. These new formulas differ from the current standard formulas in the following areas:

(1) The particle size specifications of the ingredients with the exception of dextrin are changed. (The chemical specifications of the ingredients are unchanged.)

(2) Dextrin is listed as a separate ingredient (not as a dye diluent), the quantity of which is to be controlled.

(3) The amounts of the ingredients in the formulas have been changed. The most significant changes were made in lowering the percentages of the dyes (to decrease cost) and in increasing the chlorate/bicarbonate ratios (to achieve acceptable burn times). The changes in chlorate/bicarbonate ratios are diagrammed in figures 4 to 7.

K. Cost Savings and Safety Benefits.

Using the coarse materials reduces the cost of the grenade. In addition, it reduces the possibility of fire in the processing plant and exposure of personnel to hazards. Information on these items is set forth in the following paragraphs.

1. Reduction of Dye Quantity.

During the experimental studies, approximately 140 experimental blends (nine grenades per blend) with varying quantities of ingredients were manufactured and tested to establish new formulas for each color enabling it to meet the burn-time requirements of the technical data package (TDP). The resulting formulas contained approximately 6% less dye on the average than the

Table 9. Analysis of Potassium Chlorate, 23 January 1976

Sample	Moisture	Assay	Water insoluble matter	pH of soluble matter	Hypochlorites	Chlorites	Chlorides (as KCl)	Bromates (as $KBrO_3$)	Heavy metals	Alkaline earths	Sodium salts (as $NaClO_3$)	$MgCO_3$	Particle size No.						Density
													20	60	80	100	200	325	
	%				%						%		% passing						gm/ml
MIL-P-150C, grade B, class 7	0.05 max	99.5 min	0.10 max	5-8	To pass test	–	0.10 max	0.10 max	To pass test	To pass test	0.20 max	–	–	–	99	–	90	60	–
Sample* 12	.11	99.4	.0026	6.02	Negative	–	–	.0014	Negative	Negative	.34	–	100	72	46	40	18	6	1.35

*NFX crystal-grade $KClO_3$ supplied by Hooker Chemical Co.

Table 10. Analysis of Sodium Bicarbonate, 23 January 1976

Sample	Particle size No. 20	60	80	100	200	325	Insoluble matter	Loss, in heating	Chlorides	Alkalinity	Moisture
	% (by weight) passing						%			%	
O-S-576F, CL1 specification	–	–	–	99	50-70	–	0.1[a]	35.5[b]	To pass test	98.0-100.3	0.3[a]
Sample[c] 14	100	99	48	24	1	0	Pass	36.8	Pass	100	.00

[a]Maximum.
[b]Minimum.
[c]USP grade No. 5, supplied by Church and Dwight Co.

Table 11. Analysis of Sulfur, 23 January 1976

Sample	Sulfur	Moisture	Acidity, H_2SO_4	Ash	Chlorides (as NaCl)	Sulfates (as Na_2SO_4)	Particle size No. 20	60	80	100	200	325	NH_3 and ammonium salts
	%						% passing						
MIL-S-487B specification	99.5 min	0.10 max	0.01 max	0.10 max	0.01 max	-	–	–	-	99	85-95	–	0
Sample* 11	99.9	.02	.005	.01	.005	–	75	29	21	18	9	5	Present

*16-mesh sulfur supplied by Stauffer Chemical Co.

Table 12. Proposed Formula for Red Smoke Using Coarse Chemicals and Dyes

Ingredient or specification	Particle size							Parts by weight	Tolerance
	Sieve size (μm)								
	1190	840	250	117	149	74	44		
	Sieve No.								
	16	20	60	80	100	200	325		
	% passing minimum							%	
Dye, red 9	99	–	–	–	–	15*	–	33.0	±1.0
Dextrin (MIL-D-3994)	–	–	–	99.5	–	–	–	6.0	±1.0
Sodium bicarbonate	–	–	90	–	–	5*	–	22.5	±1.5
Potassium chlorate	–	99	60	–	–	20*	–	29.5	±1.5
Sulfur	95	–	–	–	20*	–	–	9.0	±1.0

*Maximum.

Table 13. Proposed Formula for Violet Smoke Using Coarse Chemicals and Dyes

Ingredient or specification	Particle size							Parts by weight	Tolerance
	Sieve size (μm)								
	1190	840	250	177	149	74	44		
	Sieve No.								
	16	20	60	80	100	200	325		
	% passing minimum							%	
Dye, red 9	99	–	–	–	–	15*	–	7.0	±1.0
Dye, 1,4-diamino-2,3-dihydroanthraquinone	99	–	–	–	–	15*	–	28.0	±1.0
Dextrin (MIL-D-3994)	–	–	–	99.5	–	–	–	6.0	±1.0
Sodium bicarbonate	–	–	90	–	–	5*	–	23.0	±2.0
Potassium chlorate	–	99	60	–	–	20*	–	27.0	±2.0
Sulfur	85	–	–	–	20*	–	–	9.0	±1.0

*Maximum.

Table 14. Proposed Formula for Green Smoke Using Coarse Chemicals and Dyes

Ingredient or specification	Particle size							Parts by weight	Tolerance
	Sieve size (μm)								
	1190	840	250	177	149	74	44		
	Sieve No.								
	16	20	60	80	100	200	325		
	% passing minimum							%	
Dye, yellow 4	99	–	–	–	15*	–	–	3.4	±0.5
Benzanthrone	99	–	–	–	15*	–	–	7.0	±0.5
Dye, solvent green 3	99	–	–	–	15*	–	–	24.5	±1.0
Dextrin (MIL-D-3994)	–	–	–	99.5	–	–	–	5.0	±1.0
Sodium bicarbonate	–	–	90	–	–	5*	–	21.0	±2.0
Potassium chlorate	–	99	60	–	–	20*	–	30.0	±2.0
Sulfur	95	–	–	–	20*	–	–	9.0	±1.0

*Maximum.

standard formulas with correspondingly reduced quantities of dye in each grenade. The saving per grenade was as follows:

(1) Red–$0.22 per grenade

(2) Violet–$0.13 per grenade

(3) Green–$0.17 per grenade

(4) Yellow–$0.28 per grenade

Table 15. Proposed Formula For Yellow Smoke Using Coarse Chemicals and Dyes

Ingredient or specification	Particle size							Parts by weight	Tolerance
	Sieve size (µm)								
	1190	840	250	177	149	74	44		
	Sieve No.								
	16	20	60	80	100	200	325		
	% passing minimum							%	
Dye, yellow 4	99	–	–	–	15*	–	–	12.0	±0.5
Benzanthrone	99	–	–	–	15*	–	–	21.0	±1.0
Dextrin (MIL-D-3994)	–	–	–	99.5	–	--	–	6.0	±1.0
Sodium bicarbonate	–	–	90	–	–	5*	–	29.0	±2.0
Potassium chlorate	–	99	60	–	–	20*	–	23.0	±2.0
Sulfur	95	–	–	–	20*	–	–	9.0	±1.0

*Maximum.

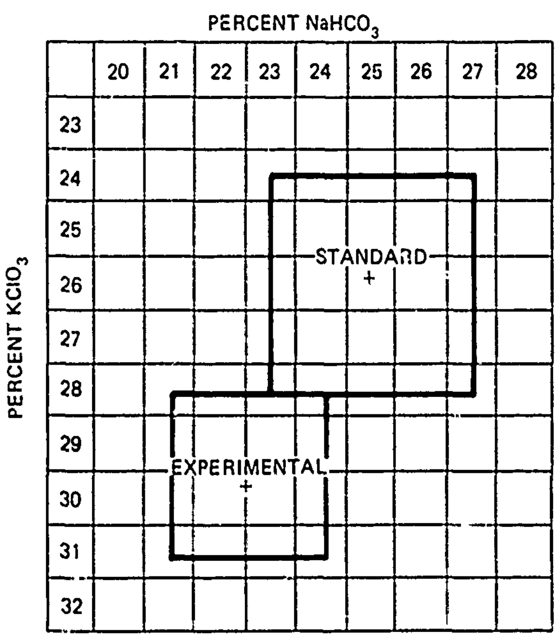

Figure 4. Diagram of $NaHCO_3$ and $KClO_3$ Percentages Allowed in Standard and Experimental Formulas–Red Smoke

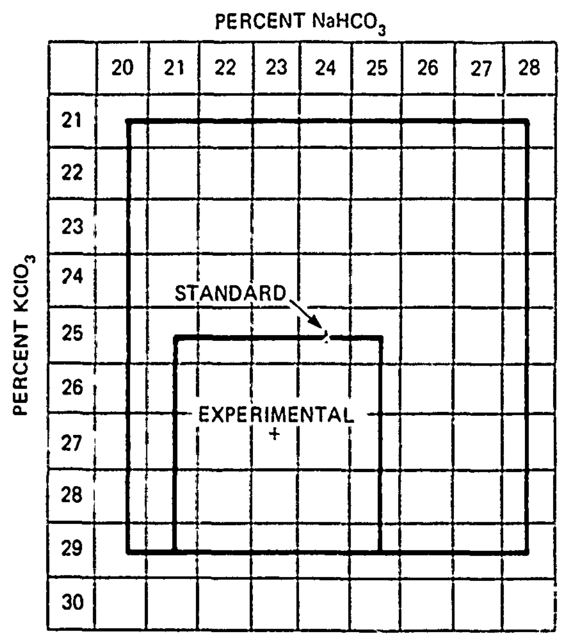

Figure 5. Diagram of $NaHCO_3$ and $KClO_3$ Percentages Allowed in Standard and Experimental Formulas–Violet Smoke

2. Increase in Cost to Process Dye.

A survey of the dye industry was conducted to determine the availability of coarse particle size dyes. This survey concluded that there was no source for the required dyes; therefore, a processing study was conducted to establish methods for agglomerating dyes. It was found that agglomeration could be accomplished on two types of equipment–the Glatt granulator and the Chilsonator. Both types of equipment agglomerate fine particle size dyes to the particle size required, at an increased manufacturing cost of $0.046 per grenade.

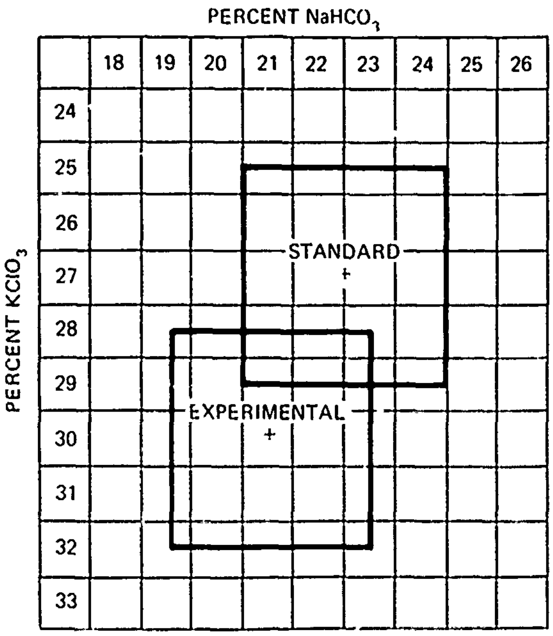

Figure 6. Diagram of $NaHCO_3$ and $KClO_3$ Percentages Allowed in Standard and Experimental Formulas–Green Smoke

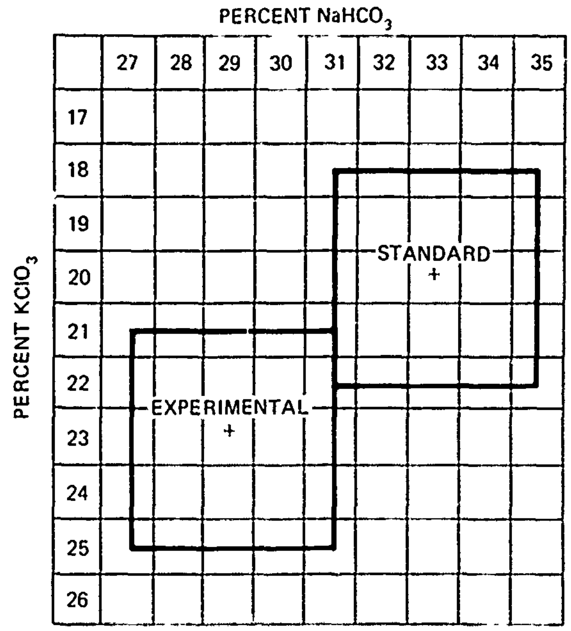

Figure 7. Diagram of $NaHCO_3$ and $KClO_3$ Percentages Allowed in Standard and Experimental Formulas–Yellow Smoke

3. Reduction of Manpower in the Pyrotechnic Mixing Facility.

a. Sieving Operations.

The current TDP material specification sets forth the use of extremely fine particle size materials in the pyrotechnic mix. Experience, over a period of many years, in manufacturing grenades at PBA has established a need for sieving the potassium chlorate, sulfur, and sodium bicarbonate prior to weighing and mixing. This operation is required for breaking up the agglomerations in the as-received material into the smooth, fine particle size required for acceptable grenades. Work with coarse particle size materials has shown that the sulfur and sodium bicarbonate do not require sieving even when exposed to a high humidity for many months. The coarse particle size potassium chlorate, however, continues to require this operation. The elimination of the sieving operation on the two chemicals reduces the manpower required at PBA by two men, which results in savings of $0.046 per grenade.

b. Mix-Drying Operation.

Currently PBA is adding acetone to the mix during the blending operation to provide an agglomerated mix that is relatively dust free and that eliminates material loss during the pressing operation. Acetone is added at the ratio of 50 pounds per 125 pounds of mix. After blending, the mix is placed on trays and dried in ovens to eliminate the acetone. Since the coarse materials result in a comparable mix, their use eliminates the need for adding acetone to the mix and also the need for the four operators in the mixing and drying facility, which results in a savings of $0.092 per grenade.

Table 16. Total Savings Per Grenade

Color	Less dye	Fewer drying operators	Fewer sieving operators	Less acetone	Subtotal	Cost to agglomerate dyes	Net savings per grenade
				$			
Red	0.220	0.092	0.046	0.045	0.403	0.046	0.357
Violet	.130	.092	.046	.045	.313	.046	.267
Green	.170	.092	.046	.045	.353	.046	.307
Yellow	.280	.092	.046	.045	.463	.046	.417

4. **Savings in Cost of Acetone.**

The elimination of the acetone itself results in a savings of approximately $0.045 per grenade.

5. **Total Savings Per Grenade.**

Total cost savings per grenade are summarized in table 16.

6. **Safety.**

The use of coarse chemicals and dyes makes it possible to blend the mix and to press grenades without using kerosene or acetone (both are allowed in the TDP). The coarse mix is free flowing and relatively dust free and can be pressed without mix loss due to airborne dusting. Although kerosene and acetone are relatively safe if properly handled, their use requires extensive auxiliary plant systems, e.g., high-rate ventilation systems to eliminate the possibilities of fire, explosion, or exposure of personnel to harmful vapors.

V. CONCLUSIONS.

(1) Coarse particle size dyes and chemicals can be used in the pyrotechnic mix of the M18 grenade.

(2) Coarse particle size sulfur, potassium chlorate, and sodium bicarbonate can be procured from commercial suppliers.

(3) Coarse particle size dyes cannot be procured from commercial suppliers.

(4) Commercial equipment that is capable of agglomerating fine particle size dyes to the required coarse particle size is available.

(5) Coarse particle size chemicals and dyes do not require the addition of kerosene or acetone for minimizing dusting during handling and pressing of the M18 grenades.

(6) Coarse particle size sulfur and sodium bicarbonate do not require sieving prior to blending operation. Coarse particle size potassium chlorate requires sieving.

VI. RECOMMENDATIONS.

It is recommended that–

(1) The M18 grenade technical data package be revised to allow the use of coarse ingredients as an alternative to the use of currently specified fine particle size ingredients.

(2) Appropriate programs be submitted to procure equipment that is capable of agglomerating fine particle size dyes into coarse particle size dyes.

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